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IS 11751 (1986): Sodium sulphite, anhydrous, photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]



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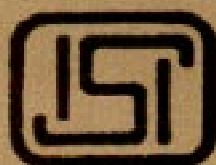
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Indian Standard

SPECIFICATION FOR
SODIUM SULPHITE, ANHYDROUS,
PHOTOGRAPHIC GRADE

UDC 661.833.522 : 771.7



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR SODIUM SULPHITE, ANHYDROUS, PHOTOGRAPHIC GRADE

Photographic Materials Sectional Committee, CDC 44

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Photographic Chemicals Subcommittee, CDC 44 : 3

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Indian Standard
**SPECIFICATION FOR
SODIUM SULPHITE, ANHYDROUS,
PHOTOGRAPHIC GRADE**

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 June 1986, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 The specification for sodium sulphite anhydrous, photographic grade was earlier covered under IS : 247-1972* along with other grades of the material. The committee responsible for formulation of this standard felt that a separate standard for the photographic grade of the material should be formulated. Consequently, this standard covering only the photographic grade of the material has been formulated and the photographic grade is being deleted from IS : 247-1972*. In this standard, a new requirement for reaction to ammoniacal silver nitrate has been included and a new *p*-nitrophenol indicator method for testing of heavy metals has been prescribed in place of litmus method as given in IS : 247-1972*.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for sodium sulphite, anhydrous, photographic grade.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of a white granular powder, free from extraneous matter.

*Specification for sodium sulphite, anhydrous (*third revision*).

†Rules for rounding off numerical values (*revised*).

2.2 Solution in Water — A freshly prepared aqueous solution of the material containing 20 g of powder in 100 ml of carbon dioxide-free water shall be colourless and free from sediment other than a slight flocculence.

2.3 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR SODIUM SULPHITE, ANHYDROUS, PHOTOGRAPHIC GRADE

(Clauses 2.3, A-5.3.1, A-6.3.1 and A-7.3.1)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Sodium sulphite (Na_2SO_3) content, percent by mass, <i>Min</i>	96.0	A-2
ii)	Insoluble matter and metals other than alkali metals, percent by mass, <i>Max</i>	0.05	A-3
iii)	Free alkali (as Na_2O), percent by mass, <i>Max</i>	0.15	A-4
iv)	Sodium thiosulphate (as $\text{Na}_2\text{S}_2\text{O}_3$), percent by mass, <i>Max</i>	0.03	A-5
v)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.002	A-6
vi)	Iron (as Fe), percent by mass, <i>Max</i>	0.005	A-7
vii)	Reaction to ammoniacal silver nitrate	To pass the test	A-8

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in suitable air-tight containers.

3.2 Marking — The containers shall be securely closed after filling and marked with the following information:

- Name of the material;
- Net mass of the material in the container;
- Manufacturer's name and his recognized trade-mark, if any;
- Batch number or lot number in code or otherwise; and
- Month and year of manufacture.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of preparation of test sample and the criteria of conformity shall be as prescribed in Appendix B.

A P P E N D I X A

(*Clause 2.3; and Table 1*)

ANALYSIS OF SODIUM SULPHITE, ANHYDROUS, PHOTOGRAPHIC GRADE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of the analysis.

A-2. DETERMINATION OF SODIUM SULPHITE

A-2.0 Principle of the Method — The material is added to a known excess of 0.1 N iodine solution. The excess iodine solution is back titrated with thiosulphate solution. The iodine consumed in oxidizing the sulphite to sulphate gives a measure of the sulphite.

A-2.1 Reagents

A-2.1.1 Standard Iodine Solution — 0.1 N (*see* IS : 2316-1968†).

A-2.1.2 Starch Indicator — Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for three minutes, allow the solution to cool and decant off the clear liquid.

*Specification for water for general laboratory use (*second revision*).

†Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

A-2.1.3 Standard Sodium Thiosulphate Solution — 0.1 N, freshly standardized (*see* IS : 2316-1968*).

A-2.2 Procedure — Weigh accurately about 0.25 g of the material in a tared weighing bottle and add it to exactly 50 ml of standard iodine solution. Allow to stand for 5 minutes and acidify with 2 ml of dilute hydrochloric acid. Titrate the excess of iodine with standard sodium thiosulphate solution using starch solution as indicator, the end point of the reaction being indicated by the disappearance of blue colour. Carry out a blank titration without using the material.

A-2.3 Calculation

$$\text{Sodium sulphite (Na}_2\text{SO}_3 \text{) content, percent by mass} = \frac{6.303 (V_1 - V_2) N}{M}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in the blank titration,

V_2 = volume in ml of standard sodium thiosulphate solution used with the material,

N = normality of standard sodium thiosulphate solution, and

M = mass in g of the material taken for the test.

A-3. DETERMINATION OF INSOLUBLE MATTER AND METALS OTHER THAN ALKALI METALS

A-3.0 Principle of the Method — Insoluble matter and metals other than alkali metals are determined by mixing the material with water, adding to the solution, ammonium oxalate, ammonium phosphate and ammonium hydroxide and weighing the residue obtained on the filter.

A-3.1 Reagents

A-3.1.1 Ammonium Oxalate Solution — 4 percent.

A-3.1.2 Ammonium Phosphate Solution — 10 percent.

A-3.1.3 Dilute Ammonium Hydroxide Solution — 10 percent (v/v).

A-3.1.4 Dilute Ammonium Hydroxide Solution — 2.5 percent (v/v).

A-3.2 Procedure — Dissolve about 10 g of the material, accurately weighed, in 75 ml of water. Add 5 ml of ammonium oxalate solution, 2 ml of ammonium phosphate solution, and 10 ml of dilute ammonium hydroxide solution (10 percent). Allow to stand overnight. Filter off

*Methods of preparation of standard solutions for colorimetric and volumetric analysis (*first revision*).

the precipitate formed through an ashless filter paper (Whatman No. 42 or equivalent) and wash with dilute ammonium hydroxide solution (2.5 percent). Dry the residue, ignite at about 600°C, cool in a desiccator and weigh.

A-3.3 Calculation

$$\begin{array}{l} \text{Insoluble matter and metals other than alkali} \\ \text{metals, percent by mass} \end{array} = 100 \frac{M_1}{M_2}$$

where

M_1 = mass in g of the ignited residue, and

M_2 = mass in g of the material taken for the test.

A-4. DETERMINATION OF FREE ALKALI

A-4.0 Principle of the Method — Sodium sulphite is oxidized to sodium sulphate by using hydrogen peroxide and the free alkali is then titrated against standard acid.

A-4.1 Reagents

A-4.1.1 Hydrogen Peroxide — approximately 30 percent (*m/v*), neutral to methyl red indicator.

A-4.1.2 Methyl Red Indicator — Dissolve 0.03 g of water soluble methyl red in water and dilute to 100 ml.

A-4.1.3 Standard Hydrochloric Acid — 0.1 N.

A-4.2 Procedure — Dissolve 5 g of the material, accurately weighed, in 50 ml of water and add 10 ml of hydrogen peroxide. Allow to stand to come to room temperature. Mix well, add a drop of methyl red indicator and titrate with standard hydrochloric acid. Run a blank without using the material.

A-4.3 Calculation

$$\begin{array}{l} \text{Free alkali (as Na}_2\text{O) ,} \\ \text{percent by mass} \end{array} = \frac{3.1 (V_1 - V_2) N}{M}$$

where

V_1 = volume in ml of standard hydrochloric acid used in the test with the material,

V_2 = volume in ml of standard hydrochloric acid used in the blank,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test.

A-5. TEST FOR SODIUM THIOSULPHATE

A-5.0 Principle of the Method — The opalescence produced by the material with potassium bromide-mercuric chloride reagent is compared with that produced by the standard sodium thiosulphate solution.

A-5.1 Apparatus

A-5.1.1 Nessler Cylinders — 50-ml capacity.

A-5.2 Reagents

A-5.2.1 Potassium Bromide-Mercuric Chloride Reagent — Dissolve 25 g each of potassium bromide and mercuric chloride in 900 ml of water at a temperature of about 50°C. Cool, dilute to 1 litre and allow to stand overnight. Filter, if necessary, to obtain a perfectly clear solution.

A-5.2.2 Standard Sodium Thiosulphate Solution — Dilute 5.0 ml of exactly 0.1 N sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$) solution (*see* A-2.1.3) to 1 litre.

A-5.3 Procedure — Dissolve 10.0 g of the material in water and dilute to exactly 100 ml. Slowly pipette out 0.5 ml of the solution into a Nessler cylinder containing 10 ml of potassium bromide-mercuric chloride reagent. In a second Nessler cylinder containing 10 ml of potassium bromide-mercuric chloride reagent, add 0.2 ml of standard sodium thiosulphate solution. Let both cylinders stand for 10 minutes without agitation. Then carefully agitate to distribute the opalescence and examine immediately (*see* Note).

NOTE — If the solutions stand for more than 15 minutes, secondary reactions may take place, vitiating the results.

A-5.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the opalescence produced with the material is not greater than that in the control test.

A-6. TEST FOR HEAVY METALS

A-6.0 Principle of the Method — The colour produced by heavy metals with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

A-6.1 Apparatus

A-6.1.1 Nessler Cylinder — 50-ml capacity.

A-6.2 Reagents

A-6.2.1 Concentrated Hydrochloric Acid — (*see* IS : 265-1976*).

*Specification for hydrochloric acid (*second revision*).

A-6.2.2 Ammonia — (see IS : 799-1978*).

A-6.2.3 Hydrogen Sulphide

A-6.2.4 Para-Nitrophenol Indicator — 0.25 percent.

A-6.2.5 Concentrated Nitric Acid — (see IS : 264-1976†).

A-6.2.6 Standard Lead Solution — Dissolve 1.60 g of lead nitrate in water, add 1 ml of concentrated nitric acid and make up the volume to 1 000 ml. Pipette 10 ml of this solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

A-6.3 Procedure — Weigh accurately about 5 g of sample in a 100-ml beaker and dissolve in 30 ml of water. Add slowly 10 ml of hydrochloric acid and evaporate the contents nearly to dryness on a water bath. Dissolve the residue in 5 ml of dilute hydrochloric acid (1 : 99) and 20 ml of water. Filter into a 100-ml volumetric flask with washing of the beaker and make up to the mark with water. Shake well and pipette 20 ml of the solution in a Nessler cylinder. Add 2 to 3 drops of *para*-nitrophenol indicator, add dilute ammonia (1 : 9) till yellow colour just disappears. Add 0.5 ml excess of ammonia. Now add 10 ml of saturated hydrogen sulphide water. Make up to 50 ml and mix well. Carry out a control test in the other Nessler cylinder using 1 ml of standard lead solution in place of the material and the same quantities of other reagents. Mix thoroughly and compare the colour produced in two Nessler cylinders.

A-6.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the brown colour produced in the test with the material is not greater than that produced in the control test.

A-7. TEST FOR IRON

A-7.0 Principle of the Method — Ferric iron gives a red colour with potassium thiocyanate. The colour produced by the material after oxidation is compared with the colour formed by a standard iron solution.

A-7.1 Apparatus

A-7.1.1 Nessler Cylinders — 50-ml capacity.

A-7.2 Reagents

A-7.2.1 Concentrated Hydrochloric Acid — conforming to IS : 265-1976‡.

A-7.2.2 Ammonium Persulphate or Potassium Persulphate

*Specification for ammonia, liquor (*first revision*).

†Specification for nitric acid (*second revision*).

‡Specification for hydrochloric acid (*second revision*).

A-7.2.3 Butanolic Potassium Thiocyanate Solution — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up the volume to 100 ml and shake vigorously until the solution is clear.

A-7.2.4 Standard Iron Solution — Dissolve 0.702 g of ferrous ammonium sulphate [$\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid (10 percent *v/v*) and dilute with water to 1 000 ml. Pipette 10 ml of the solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-7.3 Procedure — Pipette 5 ml of the solution prepared in **A-6.3** in a Nessler cylinder. Add 1 ml of concentrated hydrochloric acid, about 30 mg of ammonium persulphate or potassium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 seconds and allow the liquids to separate. Carry out a control test in another Nessler cylinder using 5 ml of standard iron solution in place of the material.

A-7.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of any red colour produced in the butanolic layer in the test with the material is not greater than that produced in the control test.

A-8. REACTION TO AMMONIACAL SILVER NITRATE

A-8.1 Reagents

A-8.1.1 Ammoniacal Silver Nitrate — Immediately before use, mix equal volumes of ammonium hydroxide (density approximately 0.91 g/ml) and 100 g/l aqueous silver nitrate solution.

A-8.2 Procedure — Prepare a blank by adding 5 ml of concentrated ammonium hydroxide to a solution of 1 g of the sample in 25 ml of water. Dissolve 1 g of the sample separately in 20 ml of water. Add 10 ml of freshly prepared reagent and mix well. Let the sample solution stand for 2 minutes and then compare with the blank.

A-8.2.1 The material shall be considered to have passed the test if the colour and turbidity produced with the sample is not more than that in the blank.

APPENDIX B

(Clause 4.1)

SAMPLING AND CRITERIA FOR CONFORMITY**B-1. GENERAL REQUIREMENTS FOR SAMPLING**

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions shall be observed.

B-1.1 Samples shall not be taken at a place exposed to weather.

B-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.4 The sample shall be placed in suitable, clean, dry and air-tight opaque glass or plastic containers.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample containers shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

B-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

B-2.2 From each lot, five containers shall be selected (see **B-2.2.1**) for drawing samples for test. In case the lot contains less than five containers, each container shall be tested.

B-2.2.1 Arrange the containers in a lot in a systematic way and starting from any container, count them as 1, 2, 3, and so on. Every r th container thus counted shall be separated for drawing for test, where

$$r = \frac{\text{Number of containers in the lot}}{5}$$

NOTE — In case, the value of r comes out to be a fractional number, it shall be taken as equal to the integral part of that value.

B-3. PREPARATION OF TEST SAMPLES

B-3.1 Drawing of Samples — Draw a representative sample from each container selected.

B-3.1.1 Mix portions of the material drawn from the same container to prepare the test sample representing the container. The quantity of material to be drawn from each container shall be sufficient to conduct all the tests given in 2.3 and shall be not less than 200 g.

B-3.1.2 The samples shall be stored in dry glass bottles or other suitable containers, sealed air-tight and labelled with all the particulars for sampling given in B-1.6.

B-3.1.3 Five test samples representing the five containers selected as in B-2.2 shall constitute a set of test samples

B-4. NUMBER OF TESTS

B-4.1 Tests for determination of sodium sulphite, heavy metals and iron shall be conducted on each of the five test samples (see B-3.1.3). Tests for all other characteristics prescribed in 2 and Table 1 shall be conducted on the composite sample prepared by mixing equal quantities of material from each of the five test samples.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For the Tests Carried Out on Five Test Samples

B-5.1.1 Sodium Sulphite — From the test results for sodium sulphite, the mean (\bar{X}) and range (R) of test results shall be calculated (range being defined as the difference between the maximum and the minimum values of test results). The lot shall be considered to have passed if the value of the expression ($\bar{X} - 0.6 R$) is equal to or greater than the respective limit prescribed in Table 1.

B-5.1.2 Free Alkali — From the test results for free alkali, the mean (\bar{X}) and range (R) of test results shall be calculated. The lot shall be considered to have passed if the value of the expression ($\bar{X} + 0.6 R$) is equal to or less than 0.1.

B-5.1.3 Heavy Metals and Iron — The lot shall be considered to have passed for the characteristics tested (see B-4.1) if each test sample passes the corresponding test prescribed.

B-5.2 For the Tests Carried Out on Composite Test Sample — The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample (see B-4.1) if the composite test sample satisfies the corresponding tests prescribed.

B-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in B-5.1 and B-5.2.